Synopsis of the Ph.D. thesis entitled

DESIGN AND DEVELOPMENT OF PROCESS TO INVESTIGATE EFFICACY OF BIOBASED SUSTAINABLE COMPOSITE MATERIAL AS ANTIFEEDANT FOR GRAIN STORAGE

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1. **Objectives**
    - Development of composite material consisting of anti-insect and pest properties and evaluation of their effectiveness in protecting stored grain.
    - To collect and segregate the waste paper and prepare pulp in the hydro pulper, followed by the development of optimized neem concentrates to be added to the pulp.
    - Examination and optimization of the effectiveness of the product in different atmospheric conditions throughout the year.
    - Evaluation of extraction process and selection of the most appropriate process based on optimization of various operating parameters.
    - The study involves material and energy balance of the process along with yield and characterization of the product.
2. **Motivation**
    - To apply recycling of paper waste to be utilized for environmental protection
    - Identification and development of innovative methods in the preservation of grain and cereals and reducing the wastage
    - Identify and understand drawbacks in existing methods of storage of grains and materials
    - Utilization of naturally renewable material as feedstock to develop eco-friendly and sustainable products
    - Cost calculation and comparative analysis
3. **Plan of work**
    - Coursework and detailed literature review and plan of experiments.
    - Neem Extraction methods based on optimization of operating parameters and yield and quality of extract
    - Preparation of composite products using neem, and paper and evaluate their effectiveness for grain storage.
    - Optimization of the application of product under different seasonal conditions
    - Cost analysis of the process and product developed
    - Organization of data, results, and discussion, and Thesis Writing.

4. Overview

The main occupation of India is Agriculture. Around 70 percent of the population earns its livelihood from agriculture. Food is the primary need of man amongst the three basic needs of man that is food, water, and shelter. India is the largest producer of commodities like cereals, pulses and oilseeds. Food grains worth Rs 7000 crores or 14 million tonnes (MT) are lost in India every year. The losses resulting from insect attacks amounted to Rs. 1300 crores (2). According to (5), insects alone are responsible for 2% or more of the post-harvest losses. Insects consume the grains while also contaminating them through their feeding activities, making them unfit for human consumption and causing financial losses.

Eco-Friendly Traditional Practices of Storing Grains

Sun drying of grains, use of ash, sand application method, plastering of storage bins with clay and cow dung, storage of pulses with common salt, turmeric application method, use garlic cloves,mixing of leaves, stepping or stamping method, use of salt and chili powder(2).

Post Harvest Losses

- Post-harvest loss is the degradation of the quality and quantity of food grains from their harvest to consumption.
- The post-harvest losses in developing country between 24 - 40 % and in developed countries the post-harvest losses are between 2-20% (9).

Factors affecting the post-harvest losses are classified as:

Abiotic factors: Moisture content and temperature, storage period, intergranular atmospheric composition, types of storage structures.

Biotic factors: Physical seed characteristics, microorganisms, insects and mites, birds, and rodents.

The known fact that each coin has two sides, likewise with production three stages of losses according to (6), are studied: pre-harvest, harvest, and post-harvest losses, each of which has serious issues. Crops that expand under the attack of insects, rust, and weeds experience pre-harvest losses. Losses incurred while harvesting are referred to as harvest losses. Post-harvest losses are those that happen both before and after the harvest but before human consumption.Favorable conditions for the growth of insects arePeriod of storage, structure of

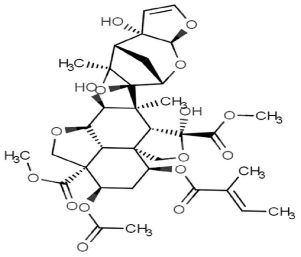
storage, temperature, moisture content, intergranular atmospheric composition, and relative humidity.

Neem Leaves Properties: Neem leaves contain the component Azadirachtin which has the insect repellent properties. Azadirachtin belongs to tetranortriterpenoid a class of limonoids,

ISO common name Not available

Other names Azadirachtin A

Chemical formula $C_{35}H_{44}O_{16}$



Molecular weight 720.71 g mol^{-1}

Melting point 154^{o}C to 158^{o}C

Maximum absorption in the ultraviolet region (UV) at 217 nm.

Hydrophilic in nature, moderately soluble in water, and highly soluble in polar organic solvents. It is non-volatile and photosensitive. Azadirachtin is the most significant active ingredient; the others are quercetin, sodium nimbinate, gedunin, salannin, nimbin, nimbidin, and nimbidol. Ascorbic acid, n-hexacosanol, amino acid, nimbin, nimbanene, 6-desacetylnimbinene, nimbandiol, nimbolide, 7-desacetyl-7-benzoylazadiradione, 7-desacetyl-7-benzoylgedunin, 17-hydroxyazadiradione, and nimbiol are present in leaves. Polyphenolic flavonoids such as quercetin and ß-sitosterol were extracted from fresh neem leaves and shown to possess antimicrobial and antifungal characteristics. The percentage of Azadirachtin in the concentrate shall be studied on the HPLC at different conditions finally resulting in the formation of pallets acting as insect repellent. The pulp is made from waste paper recycling and is converted into a composite material by mixing with Azadirachtin.Azadirachtin interferes with the growth and molting process of insects. It is responsible for the molting in insects as it interferes with the synthesis of an “ecdysteroid” hormones. Its ingestion leads to abnormal molts, growth reduction and increase mortalities.

Waste Paper

In practically every aspect of life, from domestic to commercial, paper is used. The value of recycling and reusing paper cannot be overstated. Recycling is the creation of new products from previously used materials. By safeguarding the environment's natural resources, recycling eventually saves energy. Collecting and sorting are required for recycling.

5. Scope of work

From the literature review, there is no evidence to date about the combination of waste paper pulp and neem extract for the preservation of grains. Utilizing the combination of waste paper pulp and the natural, local, cheap raw materials Neem (Azadirachtin indica)extraction combined with waste paper pulp is the innovative way to prevent the grains from loss during storage mentioned in Fig 1 below.

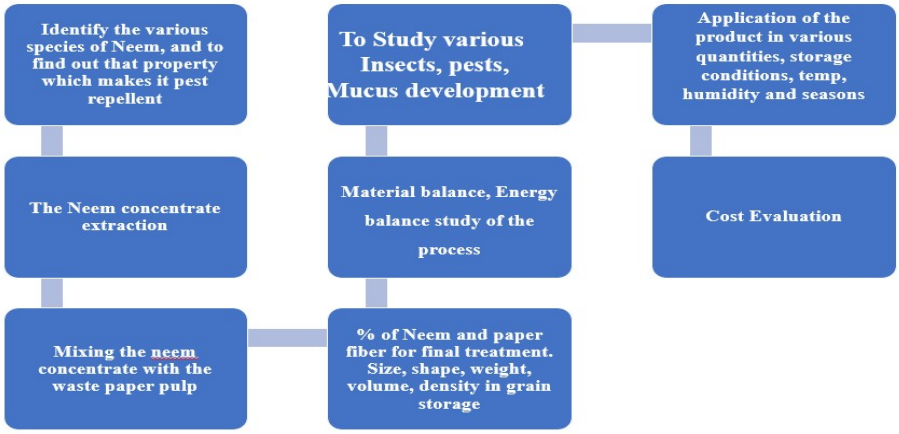

Identify the various species of Neem, and to find out that property which makes it pest repellent
To Study various Insects, pests, Mucus development
Application of the product in various quantities, storage conditions, temp, humidity and seasons
The Neem concentrate extraction
Material balance, Energy balance study of the process
Cost Evaluation
Mixing the neem concentrate with the waste paper pulp
% of Neem and paper fiber for final treatment. Size, shape, weight, volume, density in grain storage

Fig 1: Scope of work

6. Principle

Currently, various plant products are acknowledged as preferred substitutes for synthetic insecticides due to their biodegradable nature and favourable safety profiles. Recent breakthroughs in biotechnology, nanotechnology, and extraction techniques have accelerated the commercialization of plant-based products as botanical pesticides. Traditionally the neem leaves have been used for decades by spreading the leaves and the layer of grain upon the leaves simultaneously, the process is simple, and as already been proven that the neem leaves have been used for decades for the preservation of grains. However, due to the prevailing drawback of the neem leaves over time the leaves get crushed into fine pieces due to handling of grains, removal of grains for usage, and the leaves get dried with duration of storage. The

leaves get mixed with the grains during consumption or usage and offer a bitter taste to the grains resulting in the wastage of grains.

The process in Fig. 2 below is simple and can be used commercially employing the village people for the development of a biodegradable sustainable product that will benefit the farmers as well as the shopkeepers and can also find its application in the household application.

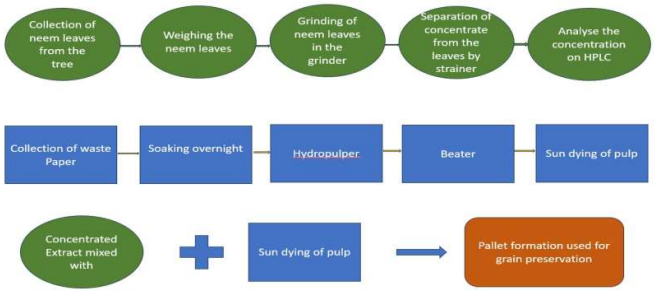

Collection of neem leaves from the tree
Weighing the neem leaves
Grinding of neem leaves in the grinder
Separation of concentrate from the leaves by strainer
Analyse the concentration on HPLC
Collection of waste Paper
Soaking overnight
Hydropulper
Beater
Sun dying of pulp
Concentrated Extract mixed with
Sun dying of pulp
Pallet formation used for grain preservation

Fig 2: Process

7. Methodology:

Table No 1: Extraction Methods

Sr No	Extraction Method	Sample 1	Sample 2
1	Distilled Water	100 gms neem leaves + 100 ml distilled water	100 gms neem leaves + concentrate from sample 1
2	Boiling Distilled Water	100 gms neem leaves 100 ml distilled water boiled at 100 degrees	100 gms neem leaves + concentrate from sample 1
3	Ethyl Alcohol	100 gms of neem leaves + 100 ml of 70% ethyl alcohol	100 gms neem leaves + concentrate from sample 1
4	Soxhlet	100 gms neem leaves powder + with di chloro methane as solvent 5 cycles were carried out for 10 min each at 25^0C.	--

The extraction of concentrate from neem leaves is represented in Fig.3. After the extraction by 4 methods mentionedin TableNo. 1, the filtrate is analyzed on HPLC as shown in Fig 4 for the detection of azadirachtin about the standard sample of azadirachtin.

Fig 3: Concentrate extraction from neem leaves

Fig 4: HPLC

Waste Paper:

- Collection of waste paper (Stationary paper, used papers from household activities) and followed by a deinking process of the paper by adding sodium hydroxide to the pulp if any ink is present on the paper.
- Soaking the paper overnight resulting in the pulp formation in the hydropulper and beater.
- After the formation of pulp in the beater by mixing water and the pulp, the pulp is simply sun dried till all the moisture in the pulp is evaporated.

Neem Pallet Formation:

- The concentrate of neem leaves and the sun-dried waste paper are mixed and the pallets are formed as shown in Fig 5.
- Testing of pallets: Size, shape, moisture, weight, density, biodegradable.
- The tablets formed as mentioned in Fig 6 can be easily separated from the grains during usage and after the use, the tablets can be simply burned in the environment posing no threat to the environment and will act as mosquito repellent.

Fig 5: Mixing of pulp and concentrate

Fig 6: Formation of Pallet

8. Findings:

Extraction

The sample trials carried out for the four extraction methods resulted in the detection of azadirachtin at the run time of 3 min which qualified the presence of azadirachtin in the samples. Physical characterization of each sample was carried out by noting its pH, colour, odour, turbidity, total dissolved solids and viscosity.

Identification of grains: The grains have been identified suchas Wheat, Chana Dal. Green Gram, and Groundnut.

A. Growth of Insects in grains:

a) Growth of insects in grains without application of insecticide

The grains 1000 gms of wheat, chana dal, green gram and groundnut were raw without any application of insecticide shall be stored in an airtight container and the observations shall be recorded.

Fig 7: Raw grains

Observation of Grains under different atmospheric conditions (Temperature and Humidity) stored from 15th July 2021 small insects were visible with the naked eye and the multiplication of insects continued till the grains were under observation till 25th September without application of any insecticide or artificial insecticide as shown in Fig 7.

b) Growth of insects in grains using neem concentrate and waste paper pulp pallets.

The grains of 1000 gms of wheat, chana dal, green gram, and groundnut with the application of neem concentrate and waste paper pulp pallets shall be stored in an airtight container and the observations shall be recorded.

Fig 8: Grains with Neem Pallets

The condition of Grains with the insertion of neem pallets under different atmospheric conditions (temperature and humidity) was studied from July 2022 to Dec 2022. It was observed that in the period of six months, there was no development of insects in the grains as shown in Fig 8.

9. **Contribution made to the existing knowledge on the subject:**

- **Novel Findings**: The thesis may present new observations that were previously unknown or not well-documented to investigate the efficacy of bio-based sustainable composite material as antifeedant for grain storage.
- **Addressing Gaps in Knowledge:** This work may fill gaps in existing literature or provide new insights into areas that were understudied or underexplored for semiconductor industry wastewater treatment.
- **Practical Applications:** The research might have implications for real-world applications or offer solutions to practical problems of grain storage.
- **Integration of Knowledge:** It might synthesize diverse sources of information and integrate knowledge from different disciplines to provide a comprehensive understanding of a complex issue.

- **Policy Implications:** The findings might have policy implications, informing decision-makers and influencing policy development to avoid losses in grain storage.
- **Generalizability:** The research might demonstrate the generalizability of findings beyond the specific case studied, contributing to broader knowledge.
- **Development of IPR:** This research contributed to the Patent publications.

10. Conclusion:

This is a multidisciplinary research work that incorporates the study of agriculture, Chemical Engineering and Chemistry, Entomology, and Environment.

This is an innovative research work including the combination of existing traditional knowledge with the neem leaves being used to protect the grains from insects. The utilization of waste paper as paper is biodegradable. The waste paper pulp in a combination of the neem leaves concentrates resulting in the formation of pallets that possess the under-stated advantages:

- Simple to separate
- Insects are repelled by neem, thus improving grain security
- Environmentally safe once utilized
- Prevention of grains acquiring a bitter taste to the grains is avoided
- Reduction in post-harvest loss, thus increasing grain production.
- Waste paper Reutilization
- Sustainable solution
- Efficient pest management system eliminating hazardous synthetic pesticides.

To date and after a prolonged literature review there is no evidence of this research conducted.

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